MINIATURE AGE DATING /MATERIALS CHARACTERIZATION INSTRUMENT FOR MARS EXPLORATION

Interim Report

JPL Task 1017

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A. OBJECTIVES

The objective is to develop a miniature age dating/material characterization instrument that will combine the most powerful dosimetric dating techniques, namely thermoluminescence (TL), optically-stimulated luminescence (OSL) and electron-spin resonance (ESR).

B. PROGRESS AND RESULTS

1. OSL Optical System Design

OSL Stimulation: We have gained experience with several Nd:YAG (2nd harmonic) laser diodes, but so far have not found one of suitable size, power and stability. Alternative stimulation sources are high-power LEDs. We have purchased several Nichia Ultra-Bright LEDs and have tested them for radiation sensitivity and stability as a function of temperature. The light output and peak wavelength have been shown to be stable (within 5% and 1 nm, respectively) for absorbed ^{60}Co gamma doses up to 500 Gy (far in excess of the doses expected to be experienced by the instrument). When operated in constant current mode (2.0 mA), the peak emission wavelength remained fixed and the output actually increased initially as the temperature was reduced.

We have used an array of Nichia diodes to successfully stimulate OSL emission from quartz. The diode array pack is shown in **Figure 1**. The array was manufactured by Risoe National Laboratory (Denmark) and consists of 6 clusters each of 6 diodes (36 LEDs in all) producing an estimated 25 mW of optical power at the sample. Figure 1 shows the array fitted with an end-window photomultiplier tube, which is unlikely to be the detector used in the final instrument.

(a) (b)

Figure 1: (a) Diode array, with photomultiplier tube; (b) diode arrays (6 x 6 clusters).

Photodetectors: We have carried out a preliminary search for potential photodetector devices, including photomultiplier tubes, P-I-N photodiodes and Avalanche photodiodes. On the basis of this, we have purchased the followed detectors:

- Hamamatsu Si Avalanche Photodiode S5343
- Hamamatsu Si P-I-N diode
- Hamamatsu H6240 side-window photomultiplier tube with integrated photon counter and power supply.

Selection of these devices was based on wavelength range, quantum efficiency and operational temperature. The devices have not yet been tested. We will conduct back-to-back tests for their wavelength dependence, efficiency and temperature dependence.

Radiation Source: To avoid the use of radioisotopes, we have decided upon an x-ray system for the radiation source in the instrument. We have identified a Moxtek "Bullet" miniature x-ray source (4W, 40 kVp) as a suitable x-ray system (**Figure 2**). This produces up to 40 keV x-rays from a Pd target. The system has not yet been tested. Performance parameters of interest include the divergence of the output beam, scattered radiation, and dose rate.

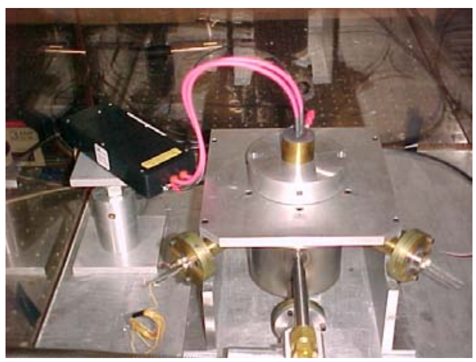

Figure 2: Moxtek x-ray unit and the low-temperature cryostat designed for laboratory characterization of the Martian simulant material

2. Characterization of Luminescence from Martian Meteorites

Our characterization program will include determining the most effective wavelengths of light to use in optical stimulation of the OSL from the simulant materials, tests for anomalous fading (signal instability), sensitivity tests, annealing temperatures, etc. With these characteristics determined at room temperature, we intend to devise measurement protocols (procedures) that will afford maximum utility for OSL stimulation and detection. We then intend to follow this with testing of the procedures at low temperature. For the latter, we have designed a test cryostat (shown in Figure 2) in which we will use the Moxtek x-ray unit.

Simulant materials: To date, we have tested samples from four Martian SNC meteorites, soil simulant JSC Mars-1, a Martian soil simulant (jeweler's rouge), and various feldspars. We have examined their basic TL and OSL properties. The studies investigated the main TL peaks, the OSL dose response curves to determine the maximum estimable radiation doses, and the ability to recover a given laboratory radiation dose (using dosimetric techniques). We have developed procedures for TL and OSL, and the following results have been obtained.

a. SNC meteorites: The TL and OSL results are summarized in Table 1. The SNC meteorites all have TL peaks ~140°C. This is a typical result for Martian meteorites with feldspar in the low-temperature, ordered state (**Figure 3, Left**). OSL dose-response experiments (**Figure 3, Right**) revealed high saturation doses for all the materials that give *theoretical* maximum estimable ages >1 My. We tested our ability to determine a previous absorbed dose by these materials (the so-called "recovered dose"). These tests showed that a laboratory radiation dose can be recovered to within 6%.

b. Feldspars: Most luminescence materials encountered on Mars will probably be of feldspathic origin. While feldpars have been used for terrestrial TL and OSL dating, these materials pose two challenges to the proposed project. Currently, no accepted single-aliquot

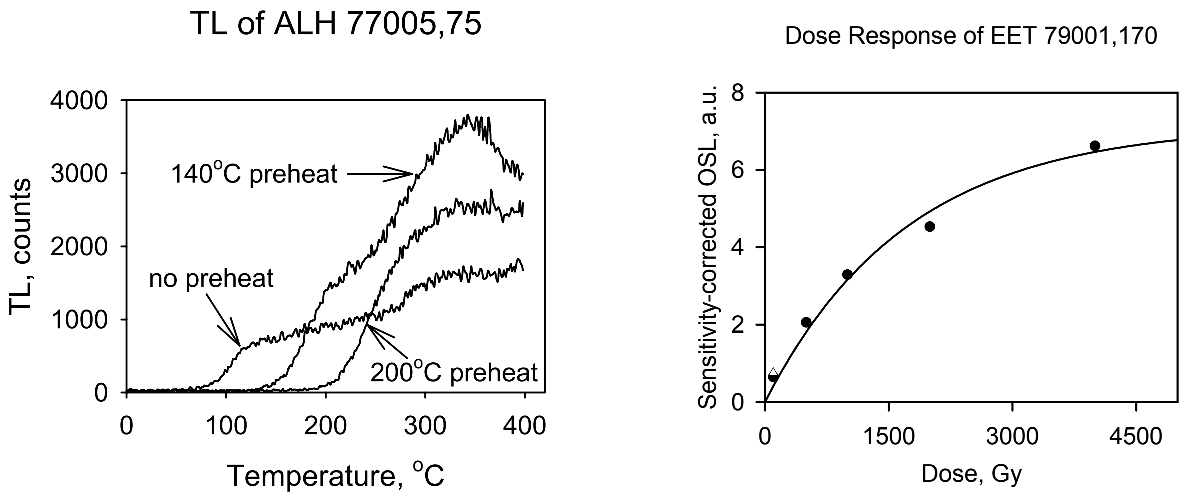

Figure 3: Left TL glowcurve of ALH 77005, 74 showing the effects of preheating. All radiation doses were 300 Gy. **Right** OSL dose response for EET 79001, 170. The open triangle represents the repeat point.

Sample	TL peaks (°C)	Max dose (Gy)	Max age (Mya)	Equiv. Dose Ratio
ALH 77005,74	~140, 340	2500	1.25	0.98
Shergotty	120, 180, 320	6300	3.15	1.04
Zagami	~150, >400	5200	2.6	1.06
EET 79001,170	~150, >400	6000	3.0	0.98
Jeweler's Rouge	140, 350	4300	2.15	0.99

Table 1: The results of TL and OSL experiments conducted on SNC meteorites and jeweler's rouge. The maximum estimable dose was found by fitting the growth curves with a saturating exponential function, and the maximum estimable age was found by assuming a dose rate of 2 $mGy.yr^{-1}$. The equivalent dose ratio is given radiation dose/ recovered radiation dose.

methods (similar to the SAR method outlined above, which was developed for quartz) exist for feldspar dating, and attempts to develop such a method have been unsuccessful (Wallinga et. al., 2000). Also, many feldpsars suffer from anomalous fading whereby thermally-stable signals nevertheless fade with time (Wintle, 1977). Although many suggestions have been made to either correct or eliminate this phenomenon (Huntley and Lamothe, 2001; Lamothe and Auclair, 1999; Vicosekas, 2000), no accepted method has been developed.

To address this issue, a project has been started to develop a single-aliquot procedure for feldspars that also eliminates anomalous fading. Once such a procedure has been developed, further work will be carried out to extend the method to polymineral samples (consisting of quartz, feldspars, and other materials) such as will be utilized for the Mars OSL dating module. The eventual goal is to use the procedure to obtain depositional ages for polymineral terrestrial samples with independent age controls.

One basic requirement for a single-aliquot technique is that repeated cycles with a fixed regeneration dose (including preheating and sensitivity-correction procedures) yield the same OSL or TL result (either the same luminescence signal or sensitivity-corrected ratio as in SAR). Five types of feldspars (microcline, anorthoclase, oligoclase, plagioclase, and andesine) have been tested for this requirement in several different ways.

The samples were first subjected to a measurement of TL to 450°C (to remove any residual signal), followed by 7 cycles of fixed regeneration dose (5 Gy), preheated to 220°C, and TL to 450°C. For all of the samples, the TL glowcurves for repeated cycles overlie each other. However, if the same experiment is conducted with OSL instead of TL measurements, the OSL decay curves do not overlie each other (i.e., there is a sensitivity change).

A subsequent experimental procedure of a measurement of TL to 450°C (to remove any residual signal), followed by 7 cycles of fixed regeneration dose (5 Gy), preheated to 220°C, OSL at 125°C for 100 s, and TL to 450°C yielded identical OSL curves with the notable exception of the first cycle (preceded by the "cleaning TL"). These results strongly imply that performing a TL measurement after the OSL measurement resets the feldspar's sensitivity, and that OSL sensitivity changes are largely governed by the availability of trapping centers (as opposed to the availability of recombination centers).

The latest experiment used the above procedure with regeneration doses ranging from 0.5-100 Gy including a repeat point of 5 Gy to construct a dose-response curve at low doses. All samples showed some non-linearity at low doses (<10 Gy), and the first cycle (after the "cleaning TL") again yielded a much higher than expected response (**Figure 4**). However, the non-linearity at low doses means that a method for sensitivity-correction still needs to be found. The increased OSL response from the first cycle is probably due to high temperature traps (> 450°C) that contribute to the OSL.

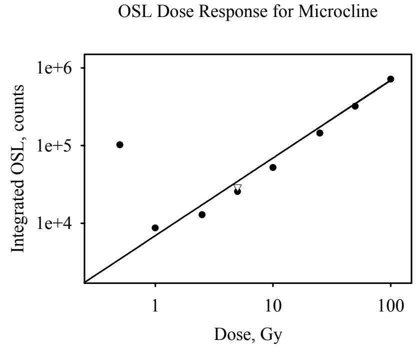

Figure 4: OSL dose-response for Microcline using procedure described in text.

3. ESR Spectrometer Design

We have designed and fabricated components of the miniature ESR spectrometer as shown in Figure 5. The frequency scan ESR spectrometer is achieved by variation of distance (total movement ~ 5 mm) between the two dielectric resonator disks ($\varepsilon_R = 29$, 1.0 cm OD x 0.55 cm ID x 0.35 cm thick) using a small DC motor (MicroMo).

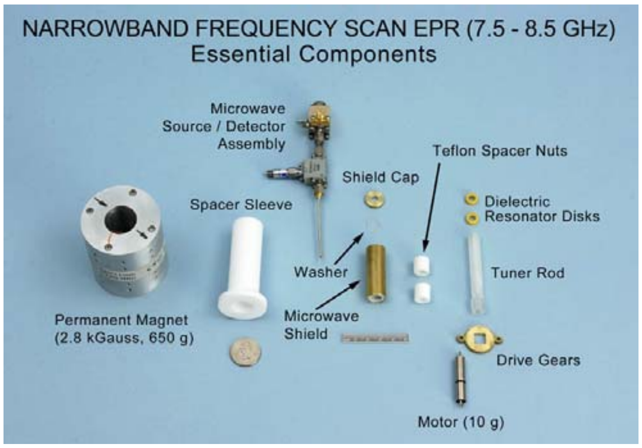

Figure 5. Essential components of the miniature ESR spectrometer that will be integrated into the Age Dating Instrument.

C. SIGNIFICANCE OF RESULTS

A method still needs to be found that fully corrects for OSL sensitivity changes in feldspars. Such a procedure will probably incorporate TL measurements to “reset” the OSL sensitivity, but a test dose correction and preheat procedures still need to be developed. The method will then be incorporated into a polymineral, single-aliquot dating technique that will subsequently be used to date terrestrial depositional events with independent age controls. Instrumentation effort will continue.

D. FINANCIAL STATUS

The total funding for this work was $110,000, of which $84,000 has been expended.

E. PERSONNEL

No other personnel were involved.

F. REFERENCES

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